

Factors Affecting Solute Analysis of Bauxite Residue Filter Cakes and Resulting Influences on Solute Recovery Calculations

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Abstract



Bauxite residue filter cakes are analyzed regularly at refineries which use filters for the final deliquoring of the solids, relying on the dry cake disposal method. Due to the different raw materials and process conditions, the bauxite residue filter cakes are different as well. Chemical analysis of bauxite residue filter cakes regarding their soluble compounds, especially total caustic, is performed routinely after forming slurry from the cake and pure water by using a certain liquid/solid (L/S) ratio. This paper shows that there is currently no cake analysis procedure which could be used without unexpected variation in the results. This is because several factors affect the analysis result at the filtration and sample preparation stages. The factors discussed in this paper include 1) the filtration and washing conditions, and the sample preparation procedure from filter cakes, encompassing 2) the L/S ratio, 3) the composition of the diluting liquid with respect to its Na₂CO₃ concentration, 4) the temperature, and 5) the cake leaching time. Additionally, examples are provided about particle size measurements and calculation of alkali recovery for the filtration stage. Significant differences in the results are observed when any detail in the sample preparation procedure is changed.

Keywords: Bauxite residue, filter cake, analysis, caustic, dissolution.

1. Introduction

Bauxite residue is the largest waste fraction generated in the alumina industry. According to the recent online statistics of World Aluminium [1], the amount of bauxite residue generated annually has increased mainly in China, and the total amount was approximately 150 million tons in 2015. The total inventory of the residue disposed of during the past decades has been estimated to be over 4 billion tons [2]. The residue is highly caustic [3], has a fine particle size distribution [4,5], and it contains a wide variety of metals, including both environmentally problematic [6] and valuable [7,8] metals. Bauxite residue has a high buffering capacity, which is largely associated with its alkaline solids content, e.g. a variety of hydroxides, carbonates, aluminates and aluminosilicates [9].

The disposal of bauxite residue in the form of filter cakes, i.e. dry cake disposal, is an emerging method, which enables disposal of the residue in landfills, instead of lagooning [10]. The total solids concentration of filtered and washed bauxite residue cakes is typically over 70 wt.% when filter presses or hyperbaric drum filters are used [11,12]. Due to the recovery of the caustic liquor from the cake in the filtration plant, the cakes are prone to a decrease of pH when the samples are prepared for the analysis of solutes by forming slurries with water. The solutes of interest are the total alkali, total caustic, sodium and alumina. All these species may leach out from the suspended solids when the cake samples are mixed with water to form suspensions for analyzing the solute concentrations, for instance by titration, atomic absorption spectrometry (AAS), or inductively

coupled plasma mass spectrometry (ICP). However, the influence of the sample preparation conditions on the analysis result has not been summarized in the existing literature.

The objective of this article is to initiate discussion on the factors affecting the analysis results of bauxite residue filter cakes. The paper summarizes the most important findings of the authors, obtained during several years of filtration research with different bauxite residues. The aim is to demonstrate, mainly by using dimensionless values, that the analysis results are highly sensitive to a number of factors in the stage of sample preparation.

2. Materials and Methods

Industrial bauxite residue samples from most continents were used as raw materials in the studies. The cakes were obtained either directly from refineries or as a result of filtration and cake washing experiments performed at Outotec Filters and Lappeenranta University of Technology, both located in Lappeenranta, Finland.

2.1. Effect of Filtration and Washing Conditions

The first part of the study was targeted at investigating the effect of filtration temperature and pressure on the leaching out of solutes from bauxite residue filter cakes. The study was performed by using two different filters, a Nutsche filter ($A \approx 20 \text{ cm}^2$) and a Larox PF 0.1 filter press ($A = 0.1 \text{ m}^2$). Cake washing was performed in both filters at $65 \text{ }^\circ\text{C}$ by using moderate wash ratios and water as the washing liquid. Additionally, leaching trials at atmospheric pressure were carried out by using the same bauxite residue slurry that was used in the filtration experiments. The results of the studies on the effect of pressure and temperature applied in the washing and solid-liquid separation stage have been presented earlier in a different form by Kinnarinen et al. [13]. For comparison, dilution of different bauxite residue *slurries* with water has also been investigated by Kinnarinen et al. [14].

2.2. Effect of Sample Preparation Procedure

The effect of the L/S ratio in the sample preparation stage was studied by using a washed, crushed and homogenized filter cake with a total solids content of 78 wt.%. In this case, the cake was obtained directly from an alumina refinery. The cake samples were reslurried and mixed with a VWR orbital shaker at the speed of 250 rpm. A wide range of liquid/solid (L/S) ratios from 1 to 49 was used. The experiments were performed at room temperature, and the samples were let to stabilize for 2 days before performing solute analyses for the clarified supernatant.

The influence of the composition of the dilution liquid was evaluated by using the same cake material as above in the L/S ratio -related investigations. The aim of this series of experiments was to study if it is possible to avoid desorption and dissolution of the suspended solids content of the cake by using a Na_2CO_3 solution with different concentrations in the cake reslurrying procedure, instead of using pure water. The Na_2CO_3 concentrations of the dilution liquids in these experiments were 25.5, 51.0 and 76.9 g/kg, and the temperatures were 20, 50 and $80 \text{ }^\circ\text{C}$. Additionally, control experiments were performed for comparison, using pure water and the same temperatures.

When a bauxite residue filter cake is suspended in water, the resulting reactions never take place immediately. To demonstrate the importance of the sample stabilization time in the analysis of the cake, experiments were performed by using short and long stabilization times (75 s and 2 d) and

mention calcium hydroxides, carbonates and aluminates, for instance tricalcium aluminate, as buffering species.

As regards the changes in the particle size distribution when bauxite residue solids are dispersed and kept mixed in water, it is obvious that the breakage of flocs plays a major role, the dissolution of particles being less important.

6. Conclusions

The aim of this paper was to present some selected results of the authors' experimental work performed with various filter cakes during the past years. All bauxite residue filter cakes have been observed to have a more or less similar behavior in the sample preparation stage prior to chemical analyses: dilution with a large amount of water makes the solute content look higher than it actually is, due to partial dissolution of the suspended solids content. When the L/S ratio used in order to re-slurry the cake for analysis has been increased from 1 to about 50, the dissolution of Na has been observed to increase by over 40 % and that of Al by over 30 %. This result depends on the refinery and the fraction of desilication products in the cake. On the other hand, the dissolution of surplus Na and Al can be prevented by diluting the cake samples with a filtrate collected from the same experiment. Increasing the pressure and temperature will inevitably increase the leaching of Na and Al from the solids. The most important factor having an influence on these dissolution phenomena is the decreasing pH of the liquid. However, a short dilution time prior to liquid analysis should not be used, because reaching the equilibrium between the solids and liquid phases may take a long time, which makes the differences between the samples become larger when the cake sample is prepared promptly. Temperature has a strong effect on the dissolution of solutes, especially when water is used as the dilution liquid, but this factor is not likely to cause significant variation in the analysis results in cases where the samples can be prepared at a constant room temperature.

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